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L1: Entry 1 of 6

File: USPT

Sep 26, 2000

US-PAT-NO: 6124391

DOCUMENT-IDENTIFIER: US 6124391 A

TITLE: Superabsorbent polymers having anti-caking characteristics

DATE-ISSUED: September 26, 2000

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Sun; Fang	Lisle	IL		
Jones; Heather S.	Greensboro	NC		
Kaiser; Thomas A.	Greensboro	NC		
Hsu; Whei-Neen	Greensboro	NC		
Molen; Ronald L.	Greensboro	NC		
Deaton; Peter A.	Greensboro	NC		
Messner; Bernfried A.	Greensboro	NC		

US-CL-CURRENT: 524/447; 523/223

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	RMIC	Draw Desc	Image
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☐ 2. Document ID: US 5686549 A

L1: Entry 2 of 6

File: USPT

Nov 11, 1997

US-PAT-NO: 5686549

DOCUMENT-IDENTIFIER: US 5686549 A

TITLE: Polymers useful in forming self-assembled bonded anisotropic ultrathin layers and their use

DATE-ISSUED: November 11, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Grainger; David W.	Beaverton	OR		
Sun; Fang	Beaverton	OR		

US-CL-CURRENT: 528/25; 425/436R, 427/407.1, 428/391, 428/400, 525/474

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	RMIC	Draw Desc	Image
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$\text{CH}_3(\text{CH}_2)_x(\text{CHOSO}_3^-\text{M}^+)\text{CH}_2\text{CH}_3$ where x and $(y+1)$ are integers of at least about 7, preferably at least about 9, and M is a water-solubilizing cation, especially sodium, unsaturated sulfates such as oleyl sulfate, the $\text{C}_{10}\text{--C}_{18}$ alkyl alkoxy sulfates ("AE,S"; especially EO 1-7 ethoxy sulfates), $\text{C}_{10}\text{--C}_{18}$ alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates), the $\text{C}_{10}\text{--C}_{18}$ glycerol ethers, the $\text{C}_{10}\text{--C}_{18}$ alkyl polyglycosides and their corresponding sulfated polyglycosides, and $\text{C}_{12}\text{--C}_{18}$ alpha-sulfonated fatty acid esters. If desired, the conventional nonionic and amphoteric surfactants such as the $\text{C}_{12}\text{--C}_{18}$ alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and $\text{C}_6\text{--C}_{12}$ alkyl phenol alkoxyates (especially ethoxylates and mixed ethoxy/propoxy), $\text{C}_{12}\text{--C}_{18}$ betaines and sulfobetaines ("sultaines"), $\text{C}_{10}\text{--C}_{18}$ amine oxides, and the like, can also be included in the overall compositions. The $\text{C}_{10}\text{--C}_{18}$ N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the $\text{C}_{12}\text{--C}_{18}$ N-methylglucamides. See WO 9,206,154. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as $\text{C}_{10}\text{--C}_{18}$ N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl $\text{C}_{12}\text{--C}_{18}$ glucamides can be used for low sudsing. $\text{C}_{10}\text{--C}_{20}$ conventional soaps may also be used. If high sudsing is desired, the branched-chain $\text{C}_{10}\text{--C}_{16}$ soaps may be used. Mixtures of anionic and nonionic surfactants are especially useful. Other conventional useful surfactants are listed in standard texts.

The preferred compositions of the present invention comprise at least about 0.01%, preferably at least 0.1%, more preferably from about 1% to about 95%, most preferably from about 1% to about 80% by weight, of an anionic detergent surfactant. Alkyl sulfate surfactants, either primary or secondary, are a type of anionic surfactant of importance for use herein. Alkyl sulfates have the general formula ROSO_3M wherein R preferably is a $\text{C}_{10}\text{--C}_{24}$ hydrocarbyl, preferably an alkyl straight or branched chain or hydroxyalkyl having a $\text{C}_{10}\text{--C}_{20}$ alkyl component, more preferably a $\text{C}_{12}\text{--C}_{18}$ alkyl or hydroxyalkyl, and M is hydrogen or a water soluble cation, e.g., an alkali metal cation (e.g., sodium, potassium, lithium), substituted or unsubstituted ammonium cations such as methyl-, dimethyl-, and trimethyl ammonium and quaternary ammonium cations, e.g., tetramethyl-ammonium and dimethyl piperdinium, and cations derived from alkanolamines such as ethanolamine, diethanolamine, triethanolamine, and mixtures thereof, and the like. Typically, alkyl chains of $\text{C}_{12}\text{--C}_{16}$ are preferred for lower wash temperatures (e.g., below about 50°C) and $\text{C}_{16}\text{--C}_{18}$ alkyl chains are preferred for higher wash temperatures (e.g., about 50°C).

Alkyl alkoxyated sulfate surfactants are another category of preferred anionic surfactant. These surfactants are water soluble salts or acids typically of the formula $\text{RO(A)}_n\text{SO}_3\text{M}$ wherein R is an unsubstituted $\text{C}_{10}\text{--C}_{24}$ alkyl or hydroxyalkyl group having a $\text{C}_{10}\text{--C}_{24}$ alkyl component, preferably a $\text{C}_{12}\text{--C}_{20}$ alkyl or hydroxyalkyl, more preferably $\text{C}_{12}\text{--C}_{18}$ alkyl or hydroxyalkyl, A is an ethoxy or propoxy unit, n is greater than zero, typically between about 0.5 and about 6, more preferably between about 0.5 and about 3, and M is hydrogen or a water soluble cation which can be, for example, a metal cation (e.g., sodium, potassium, lithium, calcium, magnesium, etc.), ammonium or substituted-ammonium cation. Alkyl ethoxylated sulfates as well as alkyl propoxylated sulfates are contemplated herein. Specific examples of substituted ammonium cations include methyl-, dimethyl-, trimethyl-ammonium and quaternary ammonium cations, such as tetramethyl-ammonium, dimethyl piperdinium and cations derived from alkanolamines,

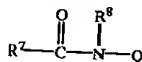
e.g., monoethanolamine, diethanolamine, and triethanolamine, and mixtures thereof. Exemplary surfactants are $\text{C}_{12}\text{--C}_{18}$ alkyl polyethoxylate (1.0) sulfate, $\text{C}_{12}\text{--C}_{18}$ alkyl polyethoxylate (2.25) sulfate, $\text{C}_{12}\text{--C}_{18}$ alkyl polyethoxylate (3.0) sulfate, and $\text{C}_{12}\text{--C}_{18}$ alkyl polyethoxylate (4.0) sulfate wherein M is conveniently selected from sodium and potassium.

The laundry detergent compositions according to the present invention may additionally comprise at least about 0.01%, preferably at least about 0.1%, more preferably at least about 1% by weight, of conventional $\text{C}_{11}\text{--C}_{18}$ alkyl benzene sulfonates ("LAS"), preferably in laundry bar embodiments and in granular laundry detergent compositions.

The preferred compositions of the present invention also comprise at least about 0.01%, preferably at least 0.1%, more preferably from about 1% to about 95%, most preferably from about 1% to about 80% by weight, of an anionic detergent surfactant. Preferred nonionic surfactants such as $\text{C}_{12}\text{--C}_{18}$ alkyl ethoxylates and $\text{C}_6\text{--C}_{12}$ alkyl phenol alkoxyates (especially ethoxylates and mixed ethoxy/propoxy), block alkylene oxide condensate of C_6 to C_{12} alkyl phenols, alkylene oxide condensates of $\text{C}_8\text{--C}_{22}$ alkanols and ethylene oxide/propylene oxide block polymers (PluronicTM-BASF Corp.), as well as semi polar nonionics (e.g., amine oxides and phosphine oxides) can be used in the present compositions. An extensive disclosure of these types of surfactants is found in U.S. Pat. No. 3,929,678, Laughlin et al., issued Dec. 30, 1975, incorporated herein by reference.

Alkylpolysaccharides such as disclosed in U.S. Pat. No. 4,565,647 Llenado (incorporated herein by reference) are also preferred nonionic surfactants in the compositions of the invention.

More preferred nonionic surfactants are the polyhydroxy fatty acid amides having the formula:



wherein R^7 is $\text{C}_5\text{--C}_{31}$ alkyl, preferably straight chain $\text{C}_7\text{--C}_{19}$ alkyl or alkenyl, more preferably straight chain $\text{C}_9\text{--C}_{17}$ alkyl or alkenyl, most preferably straight chain $\text{C}_{11}\text{--C}_{15}$ alkyl or alkenyl, or mixtures thereof; R^8 is selected from the group consisting of hydrogen, $\text{C}_1\text{--C}_4$ alkyl, $\text{C}_1\text{--C}_4$ hydroxyalkyl, preferably methyl or ethyl, more preferably methyl. Q is a polyhydroxyalkyl moiety having a linear alkyl chain with at least 3 hydroxyls directly connected to the chain, or an alkoxyated derivative thereof; preferred alkoxy is ethoxy or propoxy, and mixtures thereof. Preferred Q is derived from a reducing sugar in a reductive amination reaction. More preferably Q is a glycidyl moiety. Suitable reducing sugars include glucose, fructose, maltose, lactose, galactose, mannose, and xylose. As raw materials, high dextrose corn syrup, high fructose corn syrup, and high maltose corn syrup can be utilized as well as the individual sugars listed above. These corn syrups may yield a mix of sugar components for Q . It should be understood that it is by no means intended to exclude other suitable raw materials. Q is more preferably selected from the group consisting of $\text{---CH}_2(\text{CHOH})_n\text{CH}_2\text{OH}$, $\text{---CH}(\text{CH}_2\text{OH})(\text{CHOH})_n\text{CH}_2\text{OH}$, $\text{---CH}_2(\text{CHOH})_2\text{---}(\text{CHOR}')(\text{CHOH})\text{CH}_2\text{OH}$, and alkoxyated derivatives thereof, wherein n is an integer from 3 to 5, inclusive, and R' is hydrogen or a cyclic or aliphatic monosaccharide. Most preferred substituents for the Q moiety are glycidyls wherein n is 4, particularly $\text{---CH}_2(\text{CHOH})_4\text{CH}_2\text{OH}$.

☐ 3. Document ID: US 5686548 A

L1: Entry 3 of 6

File: USPT

Nov 11, 1997

US-PAT-NO: 5686548

DOCUMENT-IDENTIFIER: US 5686548 A

TITLE: Polymers useful in forming self-assembled bonded anisotropic ultrathin layers and their use

DATE-ISSUED: November 11, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Grainger; David W.	Beaverton	OR		
Sun; Fang	Beaverton	OR		

US-CL-CURRENT: 528/25; 425/436R, 427/407.1, 428/391, 428/400, 525/474

Full	Title	Citation	Front	Review	Classification	Date	Reference	Claims	FIGS	Draw Desc	Image
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☐ 4. Document ID: US 5686549 A

L1: Entry 4 of 6

File: EPAB

Nov 11, 1997

PUB-NO: US005686549A

DOCUMENT-IDENTIFIER: US 5686549 A

TITLE: Polymers useful in forming self-assembled bonded anisotropic ultrathin layers and their use

PUBN-DATE: November 11, 1997

INVENTOR-INFORMATION:

NAME	COUNTRY
GRAINGER, DAVID W	US
SUN, FANG	US

INT-CL (IPC): C08G 77/04

EUR-CL (EPC): G11B005/72; G11B005/72, G11B005/84

Full	Title	Citation	Front	Review	Classification	Date	Reference
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FIGS	Draw Desc	Image
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☐ 5. Document ID: US 5686548 A

L1: Entry 5 of 6

File: EPAB

Nov 11, 1997

$R^7CO-N<$ can be, for example, cocamide, stearamide, oleamide, lauramide, myristamide, capricamide, palmitamide, tallowamide, etc.

R⁸ can be, for example, methyl, ethyl, propyl, isopropyl, butyl, 2-hydroxy ethyl, or 2-hydroxy propyl.

Q can be 1-deoxyglucityl, 2-deoxyfructityl, 1-deoxymaltityl, 1-deoxylactityl, 1-deoxygalactityl, 1-deoxymannityl, 1-deoxymaltotriotityl, etc.

A particularly desirable surfactant of this type for use in the compositions herein is alkyl-N-methyl glucomide, a compound of the above formula wherein R⁷ is alkyl (preferably C₁₁-C₁₃), R⁸ is methyl and Q is 1-deoxyglucyl.

Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C₁₀-C₁₈ N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C₁₂-C₁₈ glucamides can be used for low sudsing. C₁₀-C₂₀ conventional soaps may also be used. If high sudsing is desired, the branched-chain C₁₀-C₁₆ soaps may be used.

Bleaching Compounds—Bleaching Agents and Bleach Activators

The detergent compositions herein may optionally contain bleaching agents or bleaching compositions containing a bleaching agent and one or more bleach activators. When present, bleaching agents will be at levels of from about 0.05% to about 30%, more preferably from about 1% to about 30%, most preferably from about 5% to about 20%, of the detergent composition, especially for fabric laundering. If present, the amount of bleach activators will typically be from about 0.1% to about 60%, more typically from about 0.5% to about 40% of the bleaching composition comprising the bleaching agent-plus-bleach activator.

The peroxygen bleaching compounds useful herein are those capable of yielding hydrogen peroxide in an aqueous liquor. These compounds are well known in the art and include hydrogen peroxide and the alkali metal peroxides organic peroxide bleaching compounds such as urea peroxide, and inorganic persalt bleaching compounds, such as the alkali metal perborates, percarbonates, and the like. Mixtures of two or more such bleaching compounds can also be used, if desired. Preferred peroxygen bleaching compounds include sodium perborate, commercially available in the form of mono-, tri-, and tetra-hydrate, sodium pyrophosphate peroxyhydrate, urea peroxy-hydrate, sodium peroxide, peroxyphthalate and sodium percarbonate. Particularly preferred are sodium perborate tetrahydrate, sodium perborate monohydrate and sodium percarbonate. Sodium percarbonate is especially preferred because it is very stable during storage and yet still dissolves very quickly in the bleaching liquor. It is believed that such rapid dissolution results in the formation of higher levels of percarboxylic acid and, thus, enhanced surface bleaching performance.

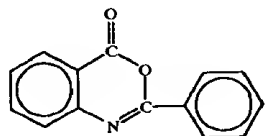
Another category of bleaching agent that can be used without restriction encompasses percarboxylic acid bleaching agents and salts thereof. Suitable examples of this class of agents include magnesium monoperoxyphthalate hexahydrate, the magnesium salt of metachloro perbenzoic acid, 4-nonylamino-4-oxoperoxybutyric acid and diperoxy-dodecanedioic acid. Such bleaching agents are disclosed in U.S. Pat. No. 4,483,781, Hartman, issued Nov. 20, 1984, U.S. patent application Ser. No. 740,446, Burns et al, filed Jun. 3, 1985, European Patent Application 0,133,354, Banks et al, published Feb. 20, 1985, and U.S. Pat. No. 4,412,934, Chung et al, issued Nov. 1, 1983. Highly preferred bleaching agents also include 6-nonylamino-6-oxoperoxyheptanoic acid as described in U.S. Pat. No. 4,634,551, issued Jan. 6, 1987 to Burns et al.

A preferred percarbonate bleach comprises dry particles having an average particle size in the range from about 500 micrometers to about 1,000 micrometers, not more than about 10% by weight of said particles being smaller than about 200 micrometers and not more than about 10% by weight of said particles being larger than about 1,250 micrometers. Optionally, the percarbonate can be coated with silicate, borate or water-soluble surfactants. Percarbonate is available from various commercial sources such as FMC, Solvay and Tokai Denka.

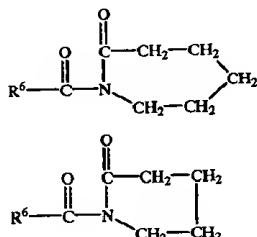
Mixtures of bleaching agents can also be used.

Peroxygen bleaching agents, the perborates, the percarbonates, etc., are preferably combined with bleach activators, which lead to the in situ production in aqueous solution (i.e., during the washing process) of the peroxy acid corresponding to the bleach activator. Various nonlimiting examples of activators are disclosed in U.S. Pat. No. 4,915, 854, issued Apr. 10, 1990 to Mao et al, and U.S. Pat. No. 4,412,934. The nonanoyloxybenzene sulfonate (NOBS) and tetraacetyl ethylene diamine (TAED) activators are typical, and mixtures thereof can also be used. See also U.S. Pat. No. 4,634,551 for other typical bleaches and activators useful herein.

Another class of bleach activators comprises the benzoxazin-type activators disclosed by Hodge et al in U.S. Pat. No. 4,966,723, issued Oct. 30, 1990, incorporated herein by reference. A highly preferred activator of the benzoxazin-type is:



Still another class of preferred bleach activators includes the acyl lactam activators, especially acyl caprolactams and acyl valerolactams of the formulae:



wherein R⁶ is H or an alkyl, aryl, alkoxyaryl, or alkaryl group containing from 1 to about 12 carbon atoms. Highly preferred lactam activators include benzoyl caprolactam, octanoyl caprolactam, 3,5,5-trimethylhexanoyl caprolactam, nonanoyl caprolactam, decanoyl caprolactam, undecenoyl caprolactam, benzoyl valerolactam, octanoyl valerolactam, decanoyl valerolactam, undecenoyl valerolactam, nonanoyl valerolactam, 3,5,5-trimethylhexanoyl valerolactam and mixtures thereof. See also U.S. Pat. No. 4,545,784, issued to Sanderson, Oct. 8, 1985, incorporated herein by reference, which discloses acyl caprolactams, including benzoyl caprolactam, adsorbed into sodium perborate.

For compositions according to the present invention comprising a bleach, preferred are peroxyacid bleaching agents,

PUB-NO: US005686548A
DOCUMENT-IDENTIFIER: US 5686548 A
TITLE: Polymers useful in forming self-assembled bonded anisotropic ultrathin layers and their use

PUBN-DATE: November 11, 1997

INVENTOR-INFORMATION:

NAME

GRAINGER, DAVID W

SUN, FANG

COUNTRY

US

US

INT-CL (IPC): C08G 77/04

EUR-CL (EPC): B05D001/18; C08G077/38, G11B005/72 , G11B005/72 , G11B005/84

Full	Title	Citation	Front	Review	Classification	Date	Reference
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☐ 6. Document ID: WO 9421386 A2

L1: Entry 6 of 6

File: EPAB

Sep 29, 1994

PUB-NO: WO009421386A2
DOCUMENT-IDENTIFIER: WO 9421386 A2
TITLE: POLYMERS USEFUL IN FORMING SELF-ASSEMBLED BONDED ANISOTROPIC ULTRATHIN LAYERS AND THEIR USE

PUBN-DATE: September 29, 1994

INVENTOR-INFORMATION:

NAME

GRAINGER, DAVID W

SUN, FANG

COUNTRY

US-CL-CURRENT: 428/421; 428/446
INT-CL (IPC): B05D 5/00; G11B 5/72; A61F 2/00; C08G 77/38; B05D 1/18
EUR-CL (EPC): G11B005/84; B05D001/18, C08G077/38 , G11B005/72 , G11B005/72

Full	Title	Citation	Front	Review	Classification	Date	Reference
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Term	Documents
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(SUN ADJ (FANG[IN])).USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	6

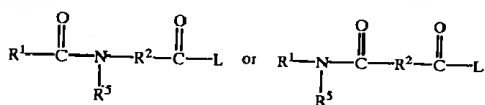
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Documents, starting with Document:

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of which amide substituted peroxyacid precursor compounds are more preferred, including those having the formula:



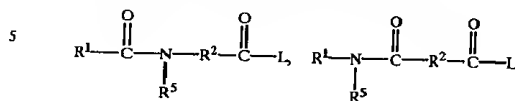
wherein R¹ is C₁-C₁₄ alkyl, aryl, alkylaryl, and mixtures thereof; R² is C₁-C₁₄ alkylene, arylene, alkylarylene, and mixtures thereof; R³ is hydrogen, C₁-C₁₀ alkyl, aryl, alkylaryl, and mixtures thereof; L is any suitable leaving group (a preferred leaving group is phenyl sulfonate). R¹ preferably contains from 6 to 12 carbon atoms. R² preferably contains from 4 to 8 carbon atoms. R¹ may contain, where applicable, branching, substitution, or both and may be sourced from either synthetic sources or natural sources including for example, tallow fat. Analogous structural variations are permissible for R². The substitution can include alkyl, halogen, nitrogen, sulphur and other typical substituent groups or organic compounds. R³ is preferably H or methyl. R¹ and R³ should not contain more than 18 carbon atoms in total. Amide substituted bleach activator compounds of this type are described in EP-A-0170386.

Preferred examples of bleach activators of the above formulae include (6-octanamido-caproyl) oxybenzenesulfonate, (6-nonanamido-caproyl) oxybenzenesulfonate, (6-decanamido-caproyl) oxybenzenesulfonate, and mixtures thereof as described in U.S. Pat. No. 4,634,551, incorporated herein by reference.

The modified polyamines of the present invention may optionally be combined with bleaching agents and bleach activator. The preferred laundry detergent compositions comprising bleaching agents comprise:

- a) at least about 0.01% to about 95% by weight, of an anionic detergent surfactant;
 - b) at least about 0.01% to about 95% by weight, of a nonionic detergent surfactant;
 - c) from about 0.01 to about 10% by weight, of a soil release polymer having effective soil release on non-cotton fabric;
 - d) from about 0.05 to about 30% by weight, of a bleach;
 - e) from about 0.05 to about 30% by weight, of a bleach activator;
 - f) from about 0.01 to about 10% by weight, a water-soluble or dispersible, bleach stable, modified polyamine cotton soil release agent according to the present invention; and
 - g) the balance carrier and adjunct ingredients.
- Still more preferably the bleach containing compositions of the present invention comprise oxygen bleaches. These oxygen bleach containing compositions comprise:
- a) at least about 0.01% to about 95% by weight, of an anionic detergent surfactant;
 - b) at least about 0.01% to about 95% by weight, of a nonionic detergent surfactant;
 - c) from about 0.01 to about 10% by weight, of a soil release polymer having effective soil release on non-cotton fabric;
 - d) optionally from about 0.05 to about 30% by weight, of a peroxygen bleach selected from the group consisting of alkali metal percarbonate, perborate, monoperphthalate, pyrophosphate peroxyhydrate, urea peroxy-hydrate and mixtures thereof;

e) optionally from about 0.05 to about 30% by weight, of a bleach activator having the formula



and mixtures thereof, wherein R¹ is C₁-C₁₄ alkyl, aryl, alkylaryl, and mixtures thereof; R² is C₁-C₁₄ alkylene, arylene, alkylarylene, and mixtures thereof; R³ is hydrogen, C₁-C₁₀ alkyl, aryl, alkylaryl, and mixtures thereof; L is any suitable leaving group;

f) from about 0.01 to about 10% by weight, a water-soluble or dispersible, bleach stable, modified polyamine cotton soil release agent according to the present invention; and

g) the balance carrier and adjunct ingredients.

Bleaching agents other than oxygen bleaching agents are also known in the art and can be utilized herein. One type of non-oxygen bleaching agent of particular interest includes photoactivated bleaching agents such as the sulfonated zinc and/or aluminum phthalocyanines. See U.S. Pat. No. 4,033,718, issued Jul. 5, 1977 to Holcombe et al. If used, detergent compositions will typically contain from about 0.025% to about 1.25%, by weight, of such bleaches, especially sulfonate zinc phthalocyanine.

If desired, the bleaching compounds can be catalyzed by means of a manganese compound. Such compounds are well known in the art and include, for example, the manganese-based catalysts disclosed in U.S. Pat. No. 5,246,621, U.S. Pat. No. 5,244,594; U.S. Pat. No. 5,194,416; U.S. Pat. No. 5,114,606; and European Pat. App. Pub. Nos. 549,271A1, 549,272A1, 544,440A2, and 544,490A1; Preferred examples of these catalysts include Mn^{II}₂(u-O)₃(1,4,7-trimethyl-1,4,7-triazacyclononane)₂(PF₆)₂, Mn^{III}₂(u-O)₃(u-OAc)₂(1,4,7-trimethyl-1,4,7-triazacyclononane)₂(ClO₄)₂, Mn^{IV}₂(u-O)₃(1,4,7-triazacyclononane)₄(ClO₄)₄, Mn^{III}Mn^{IV}₂(u-O)₃(u-OAc)₂(1,4,7-trimethyl-1,4,7-triazacyclononane)₂(ClO₄)₃, Mn^{IV}(1,4,7-trimethyl-1,4,7-triazacyclononane)-(OCH₃)₃(PF₆)₃, and mixtures thereof. Other metal-based bleach catalysts include those disclosed in U.S. Pat. No. 4,430,243 and U.S. Pat. No. 5,114,611. The use of manganese with various complex ligands to enhance bleaching is also reported in the following U.S. Pat. Nos. 4,728,455; 5,284,944; 5,246,612; 5,256,779; 5,280,117; 5,274,147; 5,153,161; and 5,227,084.

As a practical matter, and not by way of limitation, the compositions and processes herein can be adjusted to provide on the order of at least one part per ten million of the active bleach catalyst species in the aqueous washing liquor, and will preferably provide from about 0.1 ppm to about 700 ppm, more preferably from about 1 ppm to about 500 ppm, of the catalyst species in the laundry liquor.

A wide variety of other ingredients useful in detergent compositions can be included in the compositions herein, including other active ingredients, carriers, hydrotropes, processing aids, dyes or pigments, solvents for liquid formulations, solid fillers for bar compositions, etc. If high sudsing is desired, suds boosters such as the C₁₀-C₁₆ alkanolamides can be incorporated into the compositions, typically at 1%-10% levels. The C₁₀-C₁₄ monoethanol and diethanol amides illustrate a typical class of such suds boosters. Use of such suds boosters with high sudsing adjunct surfactants such as the amine oxides, betaines and sultaines noted above is also advantageous. If desired,

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USPT,PGPB,JPAB,EPAB,DWPI,TDBD	Sun, Fang [in]	6	<u>L1</u>

and are designed to address different stabilization problems depending on the type and physical form of the detergent composition.

One stabilizing approach is the use of water-soluble sources of calcium and/or magnesium ions in the finished compositions which provide such ions to the enzymes. Calcium ions are generally more effective than magnesium ions and are preferred herein if only one type of cation is being used. Typical detergent compositions, especially liquids, will comprise from about 1 to about 30, preferably from about 2 to about 20, more preferably from about 8 to about 12 millimoles of calcium ion per liter of finished detergent composition, though variation is possible depending on factors including the multiplicity, type and levels of enzymes incorporated. Preferably water-soluble calcium or magnesium salts are employed, including for example calcium chloride, calcium hydroxide, calcium formate, calcium malate, calcium maleate, calcium hydroxide and calcium acetate; more generally, calcium sulfate or magnesium salts corresponding to the exemplified calcium salts may be used. Further increased levels of Calcium and/or Magnesium may of course be useful, for example for promoting the grease-cutting action of certain types of surfactant.

Another stabilizing approach is by use of borate species. See Severson, U.S. Pat. No. 4,537,706. Borate stabilizers, when used, may be at levels of up to 10% or more of the composition though more typically, levels of up to about 3% by weight of boric acid or other borate compounds such as borax or orthoborate are suitable for liquid detergent use. Substituted boric acids such as phenylboronic acid, butaneboronic acid, p-bromophenylboronic acid or the like can be used in place of boric acid and reduced levels of total boron in detergent compositions may be possible though the use of such substituted boron derivatives.

Stabilizing systems of certain cleaning compositions may further comprise from 0 to about 10%, preferably from about 0.01% to about 6% by weight, of chlorine bleach scavengers, added to prevent chlorine bleach species present in many water supplies from attacking and inactivating the enzymes, especially under alkaline conditions. While chlorine levels in water may be small, typically in the range from about 0.5 ppm to about 1.75 ppm, the available chlorine in the total volume of water that comes in contact with the enzyme, for example during fabric-washing, can be relatively large; accordingly, enzyme stability to chlorine in-use is sometimes problematic. Since perborate or percarbonate, which have the ability to react with chlorine bleach, may present in certain of the instant compositions in amounts accounted for separately from the stabilizing system, the use of additional stabilizers against chlorine, may, most generally, not be essential, though improved results may be obtainable from their use. Suitable chlorine scavenger anions are widely known and readily available, and, if used, can be salts containing ammonium cations with sulfite, bisulfite, thiosulfite, thiosulfate, iodide, etc. Antioxidants such as carbamate, ascorbate, etc., organic amines such as ethylenediaminetetracetic acid (EDTA) or alkali metal salt thereof, monoethanolamine (MEA), and mixtures thereof can likewise be used. Likewise, special enzyme inhibition systems can be incorporated such that different enzymes have maximum compatibility. Other conventional scavengers such as bisulfate, nitrate, chloride, sources of hydrogen peroxide such as sodium perborate tetrahydrate, sodium perborate monohydrate and sodium percarbonate, as well as phosphate, condensed phosphate, acetate, benzoate, citrate, formate, lactate, malate, tartrate, salicylate, etc., and mixtures thereof can be used if desired. In general, since the

chlorine scavenger function can be performed by ingredients separately listed under better recognized functions, (e.g., hydrogen peroxide sources), there is no absolute requirement to add a separate chlorine scavenger unless a compound performing that function to the desired extent is absent from an enzyme-containing embodiment of the invention; even then, the scavenger is added only for optimum results. Moreover, the formulator will exercise a chemist's normal skill in avoiding the use of any enzyme scavenger or stabilizer which is majorly incompatible, as formulated, with other reactive ingredients, if used. In relation to the use of ammonium salts, such salts can be simply admixed with the detergent composition but are prone to adsorb water and/or liberate ammonia during storage. Accordingly, such materials, if present, are desirably protected in a particle such as that described in U.S. Pat. No. 4,652,392, Baginski et al.

The compositions herein can also optionally include one or more other detergent adjunct materials or other materials for assisting or enhancing cleaning performance, treatment of the substrate to be cleaned, or to modify the aesthetics of the detergent composition (e.g., perfumes, colorants, dyes, etc.). The following are illustrative examples of such adjunct materials.

Builders

Detergent builders can optionally be included in the compositions herein to assist in controlling mineral hardness. Inorganic as well as organic builders can be used. Builders are typically used in fabric laundering compositions to assist in the removal of particulate soils.

The level of builder can vary widely depending upon the end use of the composition and its desired physical form. When present, the compositions will typically comprise at least about 1% builder. Liquid formulations typically comprise from about 5% to about 50%, more typically about 5% to about 30%, by weight, of detergent builder. Granular formulations typically comprise from about 10% to about 80%, more typically from about 15% to about 50% by weight, of the detergent builder. Lower or higher levels of builder, however, are not meant to be excluded.

Inorganic or P-containing detergent builders include, but are not limited to, the alkali metal, ammonium and alkanolammonium salts of polyphosphates (exemplified by the triphosphates, pyrophosphates, and glassy polymeric metaphosphates), phosphonates, phytic acid, silicates, carbonates (including bicarbonates and sesquicarbonates), sulphates, and aluminosilicates. However, non-phosphate builders are required in some locales. Importantly, the compositions herein function surprisingly well even in the presence of the so-called "weak" builders (as compared with phosphates) such as citrate, or in the so-called "underbuilt" situation that may occur with zeolite or layered silicate builders.

Examples of silicate builders are the alkali metal silicates, particularly those having a $\text{SiO}_2:\text{Na}_2\text{O}$ ratio in the range 1.6:1 to 3.2:1 and layered silicates, such as the layered sodium silicates described in U.S. Pat. No. 4,664,839, issued May 12, 1987 to H. P. Rieck. NaSKS-6 is the trademark for a crystalline layered silicate marketed by Hoechst (commonly abbreviated herein as "SKS-6"). Unlike zeolite builders, the Na SKS-6 silicate builder does not contain aluminum. NaSKS-6 has the delta- Na_2SiO_5 morphology form of layered silicate. It can be prepared by methods such as those described in German DE-A-3,417,649 and DE-A-3,742,043. SKS-6 is a highly preferred layered silicate for use herein, but other such layered silicates, such as those having the general formula $\text{NaMSi}_x\text{O}_{2x+1}\cdot y\text{H}_2\text{O}$ wherein M